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DEVELOPMENT OF A PROCEDURE FOR ELECTROPLATING ON Ti-6Al-4V ALLOY

By

A. O. Schlosser and J. R. Lowery [1962] 21p 10 refs

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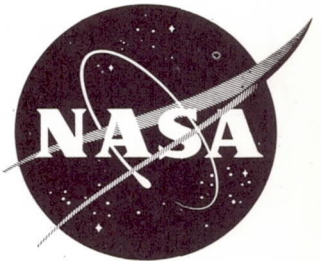
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ABSTRACT

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A procedure was developed for the production of adherent electroplated coatings on Ti-6Al-4V alloy. The process consists of anodic activation in an acetic acid-hydrofluoric acid solution, followed by electroplating by conventional methods. Coatings produced were adherent and protected the metal substrate from reactions with LOX under impact. Processing treatments did not appear to have any embrittling effects on the titanium alloy due to excessive hydrogen pick-up.

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ENGINEERING MATERIALS BRANCH
PROPULSION AND VEHICLE ENGINEERING DIVISION

TABLE OF CONTENTS

	Page
SUMMARY	1
INTRODUCTION	2
EXPERIMENTAL PROCEDURE	2
DISCUSSION OF RESULTS	3
Evaluation of Adhesion	3
Hydrogen Embrittlement	7
LOX Impact Sensitivity	8
CONCLUSIONS AND RECOMMENDATIONS	9
REFERENCES	15

LIST OF ILLUSTRATIONS

Table	Title	Page
1	Notched Tensile Tests of Ti-6Al-4V Alloy (Room Temperature)	10
2	Notched Tensile Tests of Ti-6Al-4V Alloy, -320°F (-196°C)	11
3	Bend Tests of Ti-6Al-4V Alloy (Room Temperature)	12
4	Vacuum Fusion Analyses of Ti-6Al-4V Alloy	13
5	LOX Impact Test Data on Ti-6Al-4V Alloy	14

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SUMMARY

A procedure was developed for the production of adherent electroplated coatings on Ti-6Al-4V alloy. This process consisted essentially in activation of the alloy surface by anodic treatment in a mixed acid solution, and subsequent electroplating by conventional methods. The electrolyte used for activation was composed of 875 ml of glacial acetic acid and 125 ml of 48% hydrofluoric acid. An anodic current density of 20 amps/ft² for 20 to 40 minutes and a bath temperature of 120°F (49°C) were required during the process. Copper, nickel, and chromium electroplates, as well as electroless nickel coatings, have been deposited by this method.

Conventional bend and scrape tests showed adhesion to be excellent. In addition, a small copper plated titanium alloy tank was burst by hydrostatic pressure, and another tank similarly coated was cycled between liquid nitrogen and boiling water temperatures. The adhesion of the copper to the substrate remained satisfactory in both cases. Tests conducted to detect embrittlement of the titanium alloy due to absorption of hydrogen during pretreatment and plating showed no significant differences in the mechanical properties of plated or unplated specimens. Tests for LOX impact sensitivity indicated that 0.001 inch of copper or nickel was necessary to render the titanium alloy insensitive at the 10 Kg-M (72.3 Ft-Lb) impact level.

INTRODUCTION

Because titanium alloys have higher strength-to-weight ratios at cryogenic temperatures than commonly used aluminum or steel alloys, they offer much promise for use in space vehicle systems. However, due to their reactivity with liquid oxygen (Ref. 1), they cannot be used safely in direct contact with this material. If the possibility of complete puncture of the metal substrate can be eliminated, a means of overcoming this difficulty would be to coat the titanium surfaces with materials that are LOX impact insensitive and of sufficient strength and thickness to protect the titanium alloy substrates from reaction with LOX when subjected to relatively high impacts. Electroplated coatings appeared to be useful for this application if a relatively simple method of applying a coating with consistently good adhesion were available.

The considerable weight saving and design simplification that could be achieved by mounting titanium alloy helium pressurization bottles inside the LOX tank in the S-IC stage of the Saturn V Vehicle further increased the interest in coatings for titanium. On the basis of mechanical properties at LOX temperatures and ease of fabrication, Ti-6Al-4V was chosen as the most favorable alloy for use in this application. Consequently, a program was initiated to develop a procedure for producing an adherent electroplated coating on this material.

EXPERIMENTAL PROCEDURE

From a study of the chemical properties of titanium and a knowledge of electroplating processes, it was anticipated that the main problem would be that of poor adhesion, which is caused by the formation of an extremely tenacious oxide film when titanium is exposed to the atmosphere. Before an adherent electroplated coating can be obtained, it is imperative that this film either be removed long enough to lay down an initial electrodeposit or replaced by another film that would not interfere with adhesion. After surveying the literature, several activating methods, reported to provide good adherence for electroplating on titanium, were evaluated. Work was done with chromium, nickel, electroless nickel, and copper plating, but since first indications were that more adherent coatings could be produced with copper, efforts were concentrated on plating this metal. The types of copper plating baths used included a "Coppralyte"* potassium cyanide formulation, an acid copper sulfate bath, and a copper fluoborate bath. Nickel was plated from a Watt's-type bath and chromium from a standard chromic acid-sulfate bath.

* Trade name of the E. I. du Pont de Nemours & Company

Plated specimens, 4.0 inches long by 0.5 inch wide and 0.062 inch thick, were evaluated on the basis of standard qualitative adhesion tests. The test specimens were bent repeatedly through an angle of 180° until fracture of the basis metal occurred. After fracture, attempts were made to detach the plating from the basis metal with a sharp instrument. In cases of very poor adhesion, the plating separated in large flakes or pulled off easily.

Since titanium alloys are known to be susceptible to damage from hydrogen evolved during metal finishing operations, tests were made to detect possible hydrogen embrittlement in treated specimens. Tests were made to detect changes in notch sensitivity at room temperature and at low temperatures on samples subjected to various preplating and plating processes. Some controlled bend tests and chemical analyses were also made to further verify the effects of hydrogen.

To determine LOX impact sensitivity of coated and bare specimens, sample discs 0.625 inch in diameter were immersed in LOX and impacted with a plunger at a 10 Kg-M energy level as described in Ref. 2 and Ref. 3. Visible flashes, fires, or burned areas on the specimen surface were taken as evidence of sensitivity.

DISCUSSION OF RESULTS

Evaluation of Adhesion

A number of activation processes reported to have given fair adhesion on other titanium alloys were found to be completely unsatisfactory on Ti-6Al-4V. Other reported procedures, however, showed varying degrees of success, and efforts were concentrated on improving these processes. The activation methods investigated are described in the following paragraphs. In all cases, cleaning of specimens consisted of vapor degreasing in perchlorethylene and soaking for 10 to 20 minutes in "Enthone"* alkaline cleaner #160.

1. Sodium Dichromate - Hydrofluoric Acid Etch (Ref. 4)

Panels of Ti-6Al-4V alloy, 4.0 inches long and 0.5 inch wide, were cleaned and then immersed for 20 minutes at room temperature in a bath containing 250 grams/liter of sodium dichromate and 75 grams/liter of 48% hydrofluoric acid. They were then given a conventional copper cyanide strike followed by plating in a "Coppralyte" bath. Test panels were also plated in a copper fluoborate bath after first receiving the activation treatment. The deposits had very little adhesion to the basis metal and could be peeled away easily.

* Trade name of Enthone, Incorporated

2. Acetic-Hydrofluoric Acid Etch, Immersion (Ref. 5)

Test panels were cleaned by conventional methods and immersed for 15 minutes in a bath containing 875 ml/liter of glacial acetic acid and 125 ml/liter of 48% hydrofluoric acid. They were given a copper cyanide strike and copper plated in a "Coppralyte" bath for 20 minutes. The adhesion of the deposit was completely unsatisfactory.

3. Ethylene Glycol-Hydrofluoric Acid-Zinc Fluoride Etch (Ref. 6)

This electrolytic method of activation was reported to provide good adhesion for plating of copper, chromium, nickel, tin, cadmium, and silver. Since work was concentrated on plating copper, this was the only metal tested using this technique. Process details are listed below:

- a. Degrease and clean by conventional methods.
- b. Anodically etch at 35 amps/ft² for 10 minutes in a bath containing 800 ml of ethylene glycol, 200 ml of hydrofluoric acid, and 100 grams/liter of zinc fluoride.
- c. Make cathode at 20 amps/ft² in same solution for 10 minutes.
- d. Immerse in 50% HNO₃ to dissolve loose zinc film.
- e. Rinse and plate in copper fluoborate.

In an effort to obtain better adhesion by the use of this method, a Wood's nickel strike and a copper cyanide strike were also used prior to fluoborate plating. Different current densities were employed in the etch solution along with variations in the time of treatment. The resultant deposits were uniform in appearance, but adhesion was very poor.

4. Hydrochloric Acid Etch (Ref. 7)

This process was reported to provide a surface condition on titanium base alloys receptive to a silver plate. Experience from a previous study on Ti-5Al-2.5 Sn alloy showed that satisfactory adhesion could be obtained by the use of this pretreatment. Test panels were immersed in concentrated hydrochloric acid, heated to 170°-175°F (77°-79°C) for 5-15 minutes, and then plated in both a cyanide and fluoborate copper bath. A Wood's nickel strike, applied without rinsing the specimens after the etching process, was also tried prior to copper plating. In all cases, the deposit could be peeled away from the basis metal.

5. Simultaneous Activation-Plating Treatment (Ref. 8)

This method has been employed in the plating of metals such as nickel and certain stainless steels that form passive films similar to those on titanium alloys. Panels were treated anodically at 20 amps/ft² for two minutes in a bath containing 32 oz/gal of nickel chloride and 11 fl oz/gal of hydrochloric acid. Without being removed from the bath, they next received a cathodic treatment at the same current density for six minutes. Subsequent plating was carried out by conventional methods. The quality of adhesion was improved over that of previous methods but still was not considered satisfactory.

6. Sulfuric Acid Etch

Panels were immersed for five minutes in a solution of 30% by volume of sulfuric acid in water at 220°F (104°C), followed by plating in both a cyanide and acid copper sulfate bath. The acid copper sulfate bath was also employed as a simultaneous activation-plating bath similar to the nickel chloride-hydrochloric acid process. The panels first received an anodic treatment followed by plating in the same solution. In all cases, the quality of adhesion was unsatisfactory.

7. Sulfuric Acid Pickle-Electrolytic Etch-Nickel Strike

This method consisted essentially of a combination of the 30% sulfuric acid pickle and the hydrochloric acid-nickel chloride strike. Process details were as follows:

- a. Clean by conventional methods.
- b. Water rinse.
- c. Pickle in a solution of 30% by volume of H₂SO₄ in water at a temperature of 200°F (93°C).
- d. Water rinse.
- e. Anodically etch at 25 amps/ft² in Wood's nickel strike (NiCl₂-HCl) for two minutes.
- f. Without removing from solution, make the specimen cathode at 50 amps/ft² for six minutes.
- g. Water rinse.
- h. Plate by conventional methods.

The adhesion produced by this process was inconsistent. Current density was difficult to control, and uniform etching was not achieved over the entire sample surface.

8. Ethylene Glycol-Hydrofluoric Acid (Ref. 9)

This bath, in a number of variations, was reported to provide adherent deposits of iron and copper on Ti-6Al-4V alloy. The bath which gave the best results in this study contained 800 ml of ethylene glycol and 200 ml of hydrofluoric acid (48%). The method consisted of anodically activating at 50 amps/ft² in the hydrofluoric acid-ethylene glycol mixture and then electroplating in a conventional bath. While good results from this procedure were obtained, non-adherent coatings were produced from time to time for no apparent reason. Another undesirable feature of this method was an inability to produce a uniformly activated surface. Frequently, the resultant coatings would be more adherent in the center than on the edges of test panels.

9. Acetic Acid-Hydrofluoric Acid, Electrolytic (Ref. 5)

This process as reported was used on 75A titanium alloy as an immersion etch followed by electrolytic treatment, using 60 cycle alternating current. Because an alternating current control system was not readily available, the procedure was modified slightly, and direct current was used for the electrolytic etch process. The use of this method of activation gave coatings with adhesion that was consistently good. The modified procedure consisted of an anodic etch in the mixture of acetic and hydrofluoric acid, followed by electroplating in a conventional bath. Process details are listed below:

- a. Degrease and clean by conventional methods.
- b. Anodically etch at 20 amps/ft² for 20-40 minutes at 120°F (49°C) in a bath containing 875 ml of glacial acetic acid and 125 ml of 48% hydrofluoric acid.
- c. Rinse.
- d. Electroplate by conventional methods.

Close control of this process was necessary to obtain consistently good adhesion. The temperature of the etch bath was not critical although better adhesion was obtained at a bath temperature of 120°F (49°C). Adherent coatings were produced at room temperature by increasing the etch time. It was important to avoid dilution of the etch solution by water. Studies showed that current density should be kept close to 20 amps/ft².

Of all the plating systems tried, consistently good adhesion was achieved only by using this activation method. In addition to copper, nickel and chromium were plated successfully employing this technique. Nickel was plated from a Watt's high pH (NiSO₄-NiCl₂) bath

and chromium from a conventional chromic acid-sulfate bath. Adhesion was comparable to that of copper. The good bonding qualities of this activation method were further demonstrated by applying electroless nickel coatings with adhesion that surpassed that of electroplated coatings. Electroless nickel was plated from a bath of the following composition:

Nickel Chloride - 80 oz/gal.
Sodium Hypophosphate - 1.33 oz/gal.
Sodium Citrate - 1.33 oz/gal.

As a further evaluation of the adhesion produced by the use of the acetic-hydrofluoric acid activation method and as a test of its use on larger components, two cylindrical tanks 9 inches long and 3 inches in diameter were constructed of 0.064 inch titanium sheet, plated with 1.0 mil of copper, and tested as follows: One tank was burst by hydrostatic pressure and the rupture edges were examined under magnification. There was no evidence of plating separation at these edges nor could plating be peeled or flaked off by using a sharp instrument. The second tank was thermocycled 50 times between boiling liquid nitrogen and boiling water temperatures. Adhesion remained excellent.

Plating of the cylindrical tanks for adhesion testing revealed a disadvantage in the acetic-hydrofluoric acid activation method, that being the relatively high power requirement for processing larger areas. Approximately 80 volts were required to maintain the necessary current density (20 amps/ft^2) for activating the test tanks. During activation, the temperature of the electrolyte rose from 120°F (49°C) to 180°F (82°C). It appears that the plating of large areas by this method might require the use of excessive amounts of electrical power. Further work needs to be done toward reducing the voltage requirement and concomitant electrolyte heating in order to improve the efficiency of this process.

Hydrogen Embrittlement

Since the mechanical properties of titanium alloys are adversely affected by fairly small amounts of interstitial hydrogen, it is important that the concentration of this element be controlled during processing. For most titanium alloys, more than 200 parts/million (Ref.10) of hydrogen will cause such a reduction of ductility that the metal tends to fail by brittle fracture. An acceptable activation and plating system, therefore, must provide sound adhesion to the basis metal without reducing its toughness and useful ductility by embrittlement. All procedures in which hydrogen gas is evolved at the metal surface should be suspected as sources of embrittlement.

In testing for embrittlement, tensile tests were made on notched and unnotched specimens which were activated and plated by the most promising process (acetic acid-hydrofluoric acid electrolytic etch). The notches were made prior to activating and plating of the specimen. Tables I and II show the results for room temperature and cryogenic temperature tests and indicate no significant increase in notch sensitivity. Some bend tests were also made in which specimens were supported as simple beams in a tensile machine and were bent by pressure applied at the center of the specimen. Pressure was applied continuously until a sudden reduction in load was observed, at which point the angle of bend was measured. These tests demonstrated in a qualitative way the embrittling effects of the H_2SO_4 pickle activation as compared to other processes. Results are given in Table III.

Some chemical analyses for hydrogen and oxygen content of sample specimens were made. These results are tabulated in Table IV. All of these test results showed that specimens activated in acetic-hydrofluoric acid mixture and subsequently copper plated in a fluoborate bath suffered no loss in mechanical properties or unusual absorption of hydrogen.

LOX Impact Sensitivity

LOX impact sensitivity tests were made to determine the coating thickness needed to protect adequately the titanium alloy in a liquid oxygen environment. Specimen discs, 0.625 inch in diameter and 0.062 and 0.25 inch thick, were plated with copper or nickel, using the acetic-hydrofluoric acid method of activation. Tests were conducted according to MSFC-SPEC-106, "Testing Compatibility of Materials for Liquid Oxygen Systems." Results are shown in Table V. It was found that a coating at least 0.001 inch thick of either metal was necessary to protect the titanium alloy from reaction with LOX at an impact level of 10 Kg-M (72.3 Ft-Lbs). Plated coatings 0.0005 inch thick still afforded considerable protection to the titanium alloy substrate when compared to non-plated specimens. In all cases, nickel and copper furnished the same amount of protection.

CONCLUSIONS AND RECOMMENDATIONS

By using the acetic-hydrofluoric acid anodic activation process described in this report, adherent electrodeposits of copper, nickel, and chromium can be obtained on Ti-6Al-4V alloy. Good adhesion of other electroplated deposits may reasonably be expected using this method. All other procedures which were tried yielded inconsistent or entirely unsatisfactory adhesion to the titanium alloy substrate. Electroless nickel deposits can also be applied to Ti-6Al-4V alloy with adhesion equal to that of electroplated nickel if the substrate is first activated in the acetic-hydrofluoric acid electrolyte.

No significant loss in mechanical properties, resulting from hydrogen embrittlement, occurred from the use of acetic-hydrofluoric acid activation procedure. Attempts at activation of Ti-6Al-4V by pickling in sulfuric acid resulted in a significant loss in ductility. Copper and nickel, when plated to a thickness of at least 0.001 inch on Ti-6Al-4V alloy, substantially reduced the LOX impact sensitivity of this material when tested at the 10 Kg-M level. Although both copper and nickel were shown to be equally effective in protecting a titanium alloy substrate, nickel would probably be preferred for application to vehicle hardware, e.g. the helium pressurization bottles, because of its greater hardness and scratch resistance. Before it could be considered for use, however, a more thorough study would have to be made relative to plating methods and the subsequent effect of hydrogen embrittlement.

One disadvantage found with this process was the relatively high electrical power required for large parts. This aspect will have to be investigated more fully before the process could be considered operational for use on large components. However, the primary purpose of this study was accomplished because adherent electroplated deposits were produced on titanium surfaces which reduced the LOX impact sensitivity of the titanium to an acceptable level.

TABLE I

Notched Tensile Tests of Ti-6Al-4V Alloy (Room Temperature)

Treatment	Tensile Strength KSI	Yield Strength KSI	Elongation Percent	Notched Tensile Strength KSI	Notched/ Unnotched Ratio*
Annealed	146	140	12	159	
	147	140	12	157	
	146	141	13	---	
Average	146.3	140.3	12.3	158	1.08
Solution treated and aged	163	153	10	160	
	159	154	10	150	
	158	153	10	161	
Average	160	153.3	10	157	0.98
Solution treated, aged, anodically etched in acetic-hydrofluoric acid, plated in copper fluoborate	157	151	10	158	
	157	152	9	155	
	158	153	8	153	
	155	150	9	166	
	150	146	8	168	
	155	150	9	148	
	155	150	10	156	
Average	155.3	150.3	9	157.7	1.02

* Stress concentration factor: $K_t = 10$

TABLE II

Notched Tensile Tests of Ti-6Al-4V Alloy, -320°F (-196°C)

Treatment	Tensile Strength KSI	Yield Strength KSI	Elongation Percent	Notched Tensile Strength KSI	Notched/ Unnotched Ratio*
Annealed	223	214	8	167	
	224	220	8	161	
	221	217	8	---	
	223	217	8	164	
Average	223	217	8	164	0.74
Solution treated and aged	243	233	3	149	
	240	232	3	181	
	240	233	2	166	
	241	232.6	2.7	165.3	
Average	241	232.6	2.7	165.3	0.69
Solution treated, aged, anodically etched in acetic-hydro-fluoric acid solution, fluo-borate copper plated	227	221	5	130	
	227	227	5	160	
	230	225	5	151	
	232	220	5	173	
	231	222	5	163	
	228	223	4	157	
	222	228	4	153	
	228.1	223.7	4.7	155.3	
Average	228.1	223.7	4.7	155.3	0.68

* Stress concentration factor: $K_t = 10$

TABLE IV
Vacuum Fusion Analyses of Ti-6Al-4V Alloy

Treatment	Hydrogen, PPM by Weight	Oxygen, Percent by Weight
Annealed	54	0.11
Heat treated	80	0.12
Heat treated, anodically etched in acetic-hydrofluoric acid mixture, fluoborate copper plated	82	0.12

NOTE: All values are averages of duplicate or triplicate determinations.

TABLE III

Bend Tests of Ti-6Al-4V Alloy (Room Temperature)

Treatment*	Average Bend Angle**	Remarks
None	101°	6 tests - one fracture
Anodically etched in ethylene glycol-hydrofluoric acid solution	104°	6 tests - one fracture
Anodically etched in ethylene glycol-hydrofluoric acid, copper plated in cyanide bath	84°	6 tests - four fractures
H ₂ SO ₄ pickle (220°F for 10 minutes)	41°	6 tests - all fractures
H ₂ SO ₄ pickle (220°F for 10 minutes), anodically etched in nickel strike solution, NiCl ₂ strike, copper plated in fluoborate bath	50°	6 tests - all fractures
Anodically etched in acetic-hydrofluoric acid, fluoborate copper plated	99°	4 tests - one fracture

* All samples were taken from annealed sheet.

** Outside angle measured from 180°.

TABLE V

LOX Impact Test Data on Ti-6Al-4V Alloy

Treatment	No. of Reactions	
	1/16" Thick Specimens	1/4" Thick Specimens
None	12/20 @ 72.3* 7/20 @ 36.2	18/20 @ 72.3 8/20 @ 14.0
Copper plated to a thickness of 1.0 mil	0/20 @ 72.3	0/20 @ 72.3
Copper plated to a thickness of 0.5 mil	2/20 @ 72.3	-----
Nickel plated to a thickness of 1.0 mil	0/20 @ 72.3	-----
Nickel plated to a thickness of 0.5 mil	2/20 @ 72.3	-----

* Impact energies are in Ft-Lbs. (72.3 Ft-Lbs equals 10 Kg-M)

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APPROVAL

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By

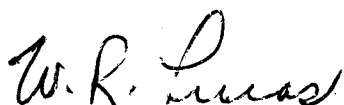
A. O. Schlosser and J. R. Lowery

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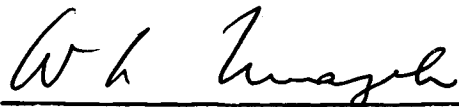
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